

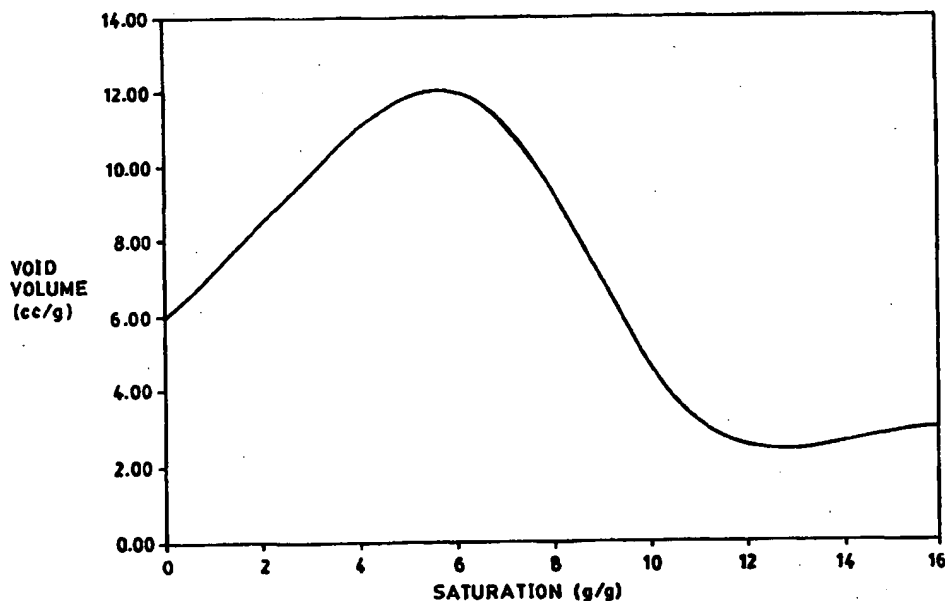
PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : A61L 15/60, A61F 13/15		A1	(11) International Publication Number: WO 99/32165
			(43) International Publication Date: 1 July 1999 (01.07.99)
(21) International Application Number: PCT/US98/26027		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).	
(22) International Filing Date: 9 December 1998 (09.12.98)			
(30) Priority Data: 60/068,534 23 December 1997 (23.12.97) US 09/192,221 16 November 1998 (16.11.98) US			
(71) Applicant: KIMBERLY-CLARK WORLDWIDE, INC. [US/US]; 401 North Lake Street, Neenah, WI 54956 (US).			
(72) Inventors: SAWYER, Lawrence, Howell; 245 Spearfield Trace, Roswell, GA 30075 (US). BURNES, Andrew, Scott; 121 Chantilly Lane, Lawrenceville, GA 30245 (US). HETZLER, Connie, Lynn; 20 Boulder Ridge Crossing, Sparta, NJ 07871 (US).		Published With international search report.	
(74) Agents: ROBINSON, James, B. et al.; Kimberly-Clark Worldwide, Inc., 401 North Lake Street, Neenah, WI 54956 (US).			

(54) Title: PULP AND SUPERABSORBENT COMPOSITE FOR IMPROVED INTAKE PERFORMANCE



(57) Abstract

There is provided an expandable absorbent composite comprising pulp in an amount from about 28 to about 90 weight percent, superabsorbent material in an amount from about 8 to about 70 weight percent and a binder fiber in an amount from about 2 to about 20 weight percent, where the composite has a density of from about 0.1 g/cc to about 0.3 g/cc. Such a composite may be used in personal care products like diapers, training pants, absorbent underpants, adult incontinence products, and the like.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand		
CM	Cameroon		Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

Pulp and Superabsorbent Composite for Improved Intake Performance

This application claims priority from US Provisional Application no. 60/068,534 filed December 23, 1997.

5

Field of the Invention

This invention relates to absorbent articles particularly absorbent structures which are useful in personal care products such as disposable sanitary napkins, diapers, or incontinence guards.

10

Background of the Invention

The desired performance objectives of personal care absorbent products include low or no leakage from the product, a dry feel to the wearer, and thinness as a means to provide comfort to the wearer. Current absorbent products, however, often fail to meet these objectives for a variety of reasons.

15

Leakage can occur, for example, due to insufficient uptake rate by layers intended to provide retention or distribution capability in the intake or target zone. Attempts to alleviate leakage occurring by this mechanism include absorbent articles that incorporate surge material structures located above (i.e., toward the wearer) the retention or distribution materials. U.S. Patent 5,364,382 to Latimer discloses nonwoven materials such as meltblowns, bonded carded webs, and pulp coforms that receive and subsequently release liquid to the retention means. The material structures of Latimer utilize large denier resilient fibers blended with small

20

25

underlying retention storage material. Additionally, Patent 5,490,846 to Ellis discloses layered structures to improve intake rates of surge materials.

Despite the development of surge materials that attempt to achieve rapid uptake and rapid release to the retention material, the objective of thinness remains to be satisfactorily reached. The cited surges are quite thick and when placed into the intake zone of the absorbent article can cause poor fit in the crotch region of the absorbent product upon initial wearing and can lead to several performance problems. Firstly, the product can leak due to gapping that is created by the bulky surge material. Secondly, the product is not comfortable to the wearer when a bulky material is utilized to provide the necessary void volume for uptake. There remains a need, therefore, for materials which will rapidly uptake an insult to the target area and either store the insult or release it for subsequent storage, and which also remain relatively thin prior to insult.

15

It is an object of this invention to provide a material for personal care products which rapidly uptakes an insult and which can store it or transfer it to an adjacent material for distribution or storage, and which remains relatively thin prior to such insult. It is another object of this invention to provide a personal care product which, prior to insult, is thin and comfortable for a wearer.

20

Summary of the Invention

The objectives of the invention are achieved by materials and products which have been designed to be very thin prior to insult and expand rapidly when insulted.

25

The invention is an expandable absorbent composite made from pulp in an amount from about 30 to about 90 weight percent, superabsorbent material in an amount from about 10 to about 70 weight percent and a binder in an effective amount, for example, from about 1 to about 20 weight percent, where the composite has a density of from about 0.1 g/cc to about 0.3 g/cc.

Such a composite may be used in personal care products like diapers, training pants, absorbent underpants, feminine hygiene products, adult incontinence products, and the like.

Brief Description of the Figures

Figures 1, 2 and 3 are graphs of the void volume generation in various composites of this invention upon saturation.

Definitions

"Hydrophilic" describes fibers or the surfaces of fibers which are wetted by the aqueous liquids in contact with the fibers. The degree of wetting of the materials can, in turn, be described in terms of the contact angles and the surface tensions of the liquids and materials involved. Equipment and techniques suitable for measuring the wettability of particular fiber materials or blends of fiber materials can be provided by a Cahn SFA-222 Surface Force Analyzer System, or a substantially equivalent system. When measured with this system, fibers having contact angles less than 90° are designated "wetable" or hydrophilic, while fibers

having contact angles equal to or greater than 90° are designated "nonwetable" or hydrophobic.

"Layer" when used in the singular can have the dual meaning of a single element or a plurality of elements.

5 "Liquid" means a nongaseous, nonparticulate substance and/or material that flows and can assume the interior shape of a container into which it is poured or placed.

As used herein the term "nonwoven fabric or web" means a web having a structure of individual fibers or threads which are interlaid, but not in an identifiable
10 manner as in a knitted fabric. Nonwoven fabrics or webs have been formed from many processes such as for example, meltblowing processes, spunbonding processes, and bonded carded web processes. The basis weight of nonwoven fabrics is usually expressed in ounces of material per square yard (osy) or grams per square meter (gsm) and the fiber diameters useful are usually expressed in
15 microns. (Note that to convert from osy to gsm, multiply osy by 33.91).

As used herein the term "microfibers" means small diameter fibers having an average diameter not greater than about 75 microns, for example, having an average diameter of from about 0.5 microns to about 50 microns, or more particularly, microfibers may have an average diameter of from about 2 microns to
20 about 40 microns. Another frequently used expression of fiber diameter is denier, which is defined as grams per 9000 meters of a fiber and may be calculated as fiber diameter in microns squared, multiplied by the density in grams/cc, multiplied by 0.00707. A lower denier indicates a finer fiber and a higher denier indicates a thicker or heavier fiber. For example, the diameter of a polypropylene fiber given as
25 15 microns may be converted to denier by squaring, multiplying the result by .89 g/cc and multiplying by .00707. Thus, a 15 micron polypropylene fiber has a denier

of about 1.42 ($15^2 \times 0.89 \times .00707 = 1.415$). Outside the United States the unit of measurement is more commonly the "tex", which is defined as the grams per kilometer of fiber. Tex may be calculated as denier/9.

"Spunbonded fibers" refers to small diameter fibers which are formed by
5 extruding molten thermoplastic material as filaments from a plurality of fine, usually circular capillaries of a spinneret with the diameter of the extruded filaments then being rapidly reduced as by, for example, in US Patent 4,340,563 to Appel et al., and US Patent 3,692,618 to Dorschner et al., US Patent 3,802,817 to Matsuki et al., US Patents 3,338,992 and 3,341,394 to Kinney, US Patent 3,502,763 to Hartman,
10 and US Patent 3,542,615 to Dobo et al. Spunbond fibers are generally not tacky when they are deposited onto a collecting surface. Spunbond fibers are generally continuous and have average diameters (from a sample of at least 10) larger than 7 microns, more particularly, between about 10 and 20 microns.

"Meltblown fibers" means fibers formed by extruding a molten thermoplastic
15 material through a plurality of fine, usually circular, die capillaries as molten threads or filaments into converging high velocity, usually hot, gas (e.g. air) streams which attenuate the filaments of molten thermoplastic material to reduce their diameter, which may be to microfiber diameter. Thereafter, the meltblown fibers are carried by the high velocity gas stream and are deposited on a collecting surface to form a
20 web of randomly disbursed meltblown fibers. Such a process is disclosed, for example, in US Patent 3,849,241. Meltblown fibers are microfibers which may be continuous or discontinuous, are generally smaller than 10 microns in average diameter, and are generally tacky when deposited onto a collecting surface.

As used herein, the term "coform" means a process in which at least one
25 meltblown diehead is arranged near a chute through which other materials are added to the web while it is forming. Such other materials may be wood pulp,

superabsorbent particles, cellulose or staple fibers, for example. Coform processes are shown in commonly assigned US Patents 4,818,464 to Lau and 4,100,324 to Anderson et al. Webs produced by the coform process are generally referred to as coform materials.

5 "Conjugate fibers" refers to fibers which have been formed from at least two polymer sources extruded from separate extruders but spun together to form one fiber. Conjugate fibers are also sometimes referred to as multicomponent or bicomponent fibers. The polymers are usually different from each other though conjugate fibers may be monocomponent fibers. The polymers are arranged in
10 substantially constantly positioned distinct zones across the cross-section of the conjugate fibers and extend continuously along the length of the conjugate fibers. The configuration of such a conjugate fiber may be, for example, a sheath/core arrangement wherein one polymer is surrounded by another or may be a side by side arrangement, a pie arrangement or an "islands-in-the-sea" arrangement.

15 Conjugate fibers are taught, for example, in US Patent 5,382,400 to Pike et al. and may be used to produce crimp in the fibers by using the differential rates of expansion and contraction of the two (or more) polymers. Such fibers may also be splittable. Crimped fibers may also be produced by mechanical means and by the process of German Patent DT 25 13 251 A1. For two component fibers, the
20 polymers may be present in ratios of 75/25, 50/50, 25/75 or any other desired ratios. The fibers may also have shapes such as those described in US Patents 5,277,976 to Hogle et al. which describes fibers with unconventional shapes.

The methods for making conjugate fibers are well known and need not be described herein in detail. To form a conjugate fiber, generally, two polymers are
25 extruded separately and fed to a polymer distribution system where the polymers are introduced into a segmented spinneret plate. The polymers follow separate paths to the

fiber spinneret and are combined in a spinneret hole which comprises either two or more concentric circular holes thus providing a sheath/core type fiber or a circular spinneret hole divided along a diameter into two parts to provide a side-by-side type fiber. The combined polymer filament is then cooled, solidified and drawn, generally by a

5 mechanical rolls system, to an intermediate filament diameter and collected. Subsequently, the filament is "cold drawn", at a temperature below its softening temperature, to the desired finished fiber diameter and is crimped/textured and cut into a desirable fiber length. Conjugate fibers can be cut into relatively short lengths, such as staple fibers which generally have lengths in the range of 25 to 51 millimeters (mm) and

10 short-cut fibers which are even shorter and generally have lengths less than 18 millimeters. See, for example, U.S. Patent No. 4,789,592 to Taniguchi et al. and U.S. Patent No. 5,336,552 to Strack et al, both of which are incorporated herein by reference in their entirety.

"Bonded carded web" refers to webs that are made from staple fibers which

15 are sent through a combing or carding unit, which breaks apart and aligns the staple fibers in the machine direction to form a generally machine direction-oriented fibrous nonwoven web. Such fibers are usually purchased in bales which are placed in a picker which separates the fibers prior to the carding unit. Once the web is formed, it then is bonded by one or more of several known bonding methods. One such

20 bonding method is powder bonding, wherein a powdered adhesive is distributed through the web and then activated, usually by heating the web and adhesive with hot air. Another suitable bonding method is pattern bonding, wherein heated calender rolls or ultrasonic bonding equipment are used to bond the fibers together, usually in a localized bond pattern, though the web can be bonded across its entire

25 surface if so desired. Another suitable and well-known bonding method, particularly when using conjugate staple fibers, is through-air bonding.

"Airlaying" is a well known process by which a fibrous nonwoven layer can be formed. In the airlaying process, bundles of small fibers having typical lengths ranging from about 2 to about 19 millimeters (mm) are separated and entrained in an air supply and then deposited onto a forming screen, usually with the assistance of a vacuum supply. The randomly deposited fibers then are bonded to one another using, for example, hot air or a spray adhesive.

"Personal care product" means diapers, training pants, absorbent underpants, feminine hygiene products and adult incontinence products.

10

Test Methods

Material caliper (thickness): The caliper of materials, which is a measure of thickness, is measured at 0.05 psi with a Starret-type bulk tester, in units of centimeters.

15 Density: The density of the materials is calculated by dividing the weight per unit area of a sample in grams per square meter (gsm) by the caliper of the sample in millimeters (mm) at 0.05 psi (68.9 Pascals) and multiplying the result by 0.001 to convert the value to grams per cubic centimeter (g/cc). A total of three samples would be evaluated and averaged for the density values.

20

Absorption Time Index (ATI): In this test the absorbent capacity of a superabsorbent material is determined versus time for up to 200 minutes under light pressure, e.g. about 0.01 psi.

25 A one inch (25.4 mm) inside diameter cylinder with an integral 100 mesh stainless steel screen on one end is used to hold 0.16 ± 0.005 grams of dry superabsorbent. The superabsorbent should be carefully placed in the cylinder so

that superabsorbent does not stick to the sides of the cylinder. The cylinder should be tapped gently to more evenly distribute the superabsorbent on the screen. A 4.4 gram, 0.995 diameter plastic piston is then placed in the cylinder and the cylinder, piston and superabsorbent assembly weighed. The assembly is placed in a 3 inch
5 by 3 inch (76.4 mm by 76.4 mm) fluid basin having a 0.875 weight percent NaCl saline solution to a depth of 1 cm. Tap the cylinder gently to remove any air trapped under it and maintain the saline solution depth at 1 cm throughout the test.

Use a timer capable of reading 200 minutes in one second intervals. Start the timer and after 5 minutes in the solution, remove the assembly and blot on
10 absorbent paper. A preferred paper is Kleenex® Premium Dinner Napkins from Kimberly-Clark Corp. though any other effective paper may be used. In blotting, press the paper tightly against the cylinder to ensure good contact. Touch the cylinder three times to dry paper and there should be very little liquid removed the third time. Weigh the assembly and return assembly to the fluid basin. Blotting and
15 weighing should take about 5 seconds and the timer should be kept running throughout the test. Take readings at 5, 10, 15, 30, 45, 60, 75, 90, 120, 160 and 200 minutes. Use fresh dry napkins for each reading.

After the final reading, calculate the grams of fluid absorbed per gram of superabsorbent. The amount of liquid absorbed at particular times divided by the
20 amount absorbed at 200 minutes may be plotted versus time for a graphical representation of the absorption rate.

The ATI is calculated as follows:

$$ATI = (t_{10} + t_{20} + t_{30} + t_{40} + t_{50} + t_{60} + t_{70} + t_{80} + t_{90}) / 9$$

where t_n is the time in minutes at which n percentage of the absorbent
25 capacity at 200 minutes is used, e.g. t_{30} is the time at which 30 percent of the total capacity is used.

Gravimetric Absorbency Test (GAT): This test measures the time an absorbent composite takes to absorb an insult. The void volume and mass of the sample are also measured. Place a 6.8 cm diameter circle of material to be tested
5 onto the sample platform of a GAT unit tester with the delivery point in the center of the sample. The GAT tester is available from M/K Systems, Inc., of Danvers, MA 01923 and is model no. M/K241 Serial no. G1048. After zeroing the instrument, set the pressure to 4 cm of positive hydrostatic pressure. Place a weight on the sample to provide 0.05 psi (68.9 Pascals) to the sample. Apply fluid to the sample,
10 maintaining constant hydrostatic pressure and record the time it takes to intake 10 grams of 0.875 weight percent NaCl saline solution. Allow the sample to equilibrate for 30 minutes and repeat until the desired number of insults has been delivered. After the final insult, remove the sample and measure the mass and thickness while under 0.05 psi pressure.

15

Detailed Description of the Invention

The material structures of this invention have been designed to be very thin materials that expand rapidly when insulted. Therefore, when the user applies the
20 absorbent article it is very thin and comfortable and facilitates good fit. As the material structure expands during use, it creates the necessary void volume to accommodate incoming fluid which in turn reduces the chances for leakage.

There are several embodiments of this invention described below. All have
25 been designed to begin with a very thin structure which then expands upon contact with the insult fluid.

The material structure consists of modified pulp fiber, modified superabsorbent, and a small amount of binder, preferably thermally activated binder fiber. The material is initially produced and stabilized at a low density. The material is then compressed and fixed at a higher density as part of an absorbent system that meets the thinness requirements. When insulted, the compressed structure recovers a substantial portion of its as produced caliper, achieving an intermediate density between as produced and compressed states. The expandable composite provides the greatest benefit when used as part of an absorbent system that moves liquid out of the target zone.

Specific examples include structures containing pulp fiber, superabsorbent material, and thermoplastic binder fiber and were produced on a Dan Web airway system. Composites within the scope of this invention contained the Weyerhaeuser Company's NHB-416 crosslinked pulp fiber at 75% or 55% by weight, and conjugate fibers by the Hoechst -Celanese Company called Celbond T105 at 5% by weight. Superabsorbent materials were Dow Chemical's AFA-130-53C, Stockhausen's W77553 or Stockhausen's FAV880A at 20% or 40% by weight. Materials were produced at low density (0.05 g/cc) and moderate density (0.1 g/cc) and thermally stabilized on line. Stockhausen's W77553 is a bulk polymerized polyacrylate with a hydrophobic surface treatment. Stockhausen's FAV880A is commercially available from the Stockhausen Company of Greensboro, NC 27406 and is a highly crosslinked surface superabsorbent. AFA 130-53C is a 850 to 1400 micron suspension polymerized polyacrylate particle from The Dow Chemical Company of Midland, MI. Composites were compacted to the absorbent product target density of 0.20 g/cc by hot pressing in a shimmed carver press (80-100°C for 1 minute).

This pressing procedure resulted in materials that retained the 0.20 g/cc density over extended times. While other pressing procedures and other commercially available compressing procedures including continuous, on-line calendering, could also be utilized to produce the desired structures, lower temperature pressing (<50 °C) with and without moisture added, resulted in materials that rapidly rebulked under ambient conditions and would not maintain the desired thinness long enough to fall within the scope of this invention.

Higher temperature bonding (150 °C) resulted in samples that were over bonded and that did not rapidly expand when wetted and therefore did not result in the required void volume needed to fall within the scope of this invention. This contrasts with the sample that was bonded at better conditions (80-100 °C) and expands rapidly when wetted to greater than 80% of its uncompressed thickness and greater than 90% of its thickness when saturated if starting from the uncompressed state.

The preferred material structure, therefore, is a wet resilient but very hydrophilic, chemically cross-linked pulp fiber such as Weyerhaeuser NHB416 (28 to 90 weight percent), slow superabsorbent such as Dow AFA-130-53C or Stockhausen W77553 (8 to 70 weight percent), and a binder fiber such as sheath core conjugate fibers produced by Chisso, Hercules, Danaklon, or Hoechst - Celanese such as Celbond T105/T255 fibers.

By "slow rate" superabsorbent what is meant is a superabsorbent having an absorption time index (ATI) of at least 5 minutes and preferably more than 10 minutes. Note that though slow superabsorbents are preferred, fast or traditional

superabsorbents will also function, as discussed below. Fast superabsorbents act to bind liquid rapidly, limiting spreading and wicking and reducing the overall distribution of liquid in the absorbent product but still providing very good intake performance.

5

The binder must be present in an effective amount to hold the structure together. While lower amounts may be possible a binder amount of between about 1 and about 20 weight percent are believed effective. Conjugate binder fibers are preferred though any binder known to be effective may be used. The binder must
10 perform its function when the composite is compressed and maintain the composite in the higher density state until a liquid insult. Upon insult, the binder must allow the composite to expand to substantially its pre-compression caliper. If the binder is in fiber form it is preferable that the fibers be as fine as possible.

15 Moisture sensitive binders are available in powder, liquid, or fibrous form that may be activated using heat and/or small amounts of moisture. Binding systems may be polyvinyl alcohol adhesives, powders or fibers that dissolve in fluids. Some specific examples of polyvinyl alcohols have easily reversible crosslinks that allow changes in the adhesive property upon contact with the insult allowing the resilient
20 structure to expand. Water sensitive hot melt adhesives could also be used that have time triggers based on controlled hydrophilicity or water triggerable polymers could be used such as base sensitive acrylics. Binders also include polyacrylic amides, polyacrylic acid and its copolymers, starch binders, cellulosic binders, and protein based binders.

25

Conjugate binder fibers which may be used in the practice of this invention may be crimped. One side of the conjugate fiber may have a water triggerable first component such as polyethylene oxide while the other side of the fiber may have a resilient fiber second component such as polyethylene terephthalate (PET). Many
5 polymers are degradable in essentially plain water such as tap water which typically has a pH in the range of about 6.5 to about 8.5 and may serve as the water degradable portion of the conjugate fiber. Polymers can also be selected for the first component which are sensitive to or become degradable as a result of pH change, dissolved ion concentration change and/or temperature change in the aqueous environment.

10

Another mechanism which can be used to trigger water-degradability is ion sensitivity, where the term "ion" is given its conventional meaning of an atom or molecularly bonded group of atoms, which has gained or lost one or more electrons and consequently has a negative or positive electrical charge. Certain polymers contain
15 acid-based (R-COO⁻) components which are held together by hydrogen bonding. In a dry state, these polymers remain solid. See for example, U.S. Patent No. 4,419,403 to Varona which is incorporated herein by reference in its entirety.

Examples of polymers capable of degrading in aqueous mixtures or toilet water
20 are poly (vinyl alcohol) graft copolymers supplied by the Nippon Synthetic Chemical Co., Ltd., Osaka, Japan, coded Ecomaty AX2000, AX10000 and AX-300G. The Nippon polymers are cold water soluble but somewhat slower in their rate of solubility than the Fuller polymers. Yet another first component polymer could be a polyether block amide, coded Pebax MX1074, supplied by Atochem (USA) located in Philadelphia,
25 Pennsylvania. The Pebax MX1074 polymer is composed of epsilon-caprolactam (Nylon 12) and tetramethylene glycol monomers. These monomers are polymerized to make a

series of polyether block amide copolymers. The Pebax polymer is not water soluble but is water-swellable, and therefore could also be used in a higher water volume environment as well. The Fuller polymers can be matched to a second component (core) polymer with a softening or melting temperature at least about 10°C higher, such as would be the case with polypropylene. The Nippon or Atochem polymers can be matched with a higher melting temperature range second component polymer such as polypropylene or poly (butylene terephthalate).

More particularly, the expandable absorbent composite of this invention may have pulp in an amount from about 30 to about 80 weight percent, superabsorbent in an amount from about 10 to about 60 weight percent and binder in an amount from about 1 to about 10 weight percent. Still more particularly, the expandable absorbent composite may have pulp in an amount from about 55 to about 75 weight percent, superabsorbent in an amount from about 20 to about 40 weight percent and binder in an amount from about 3 to about 8 weight percent. Such composites must have a density after compression and before wetting of between about 0.1 g/cc and 0.30 g/cc, and preferably about 0.20 g/cc.

The attached Figures show the generation of void volume as a function of saturation for three material structures that fall within the scope of this invention. In the Figures, void volume is shown on the Y axis in units of cc/g and saturation is shown on the X axis in units of g/g.

Example 1

Figure 1 is a graph of the void volume generation in a composite containing fast superabsorbent material Stockhausen FAV880 at 20 weight percent, chemically crosslinked pulp NHB416 from Weyerhaeuser at 75 weight percent, and Celbond T105 from Hoechst-Celanese at 5 weight percent upon saturation. The as-produced density was 0.04 g/cc and the sample consisted of 2 layers with a total plied basis weight of 400 gsm. The sample was pressed for 1 minute at 80 C to a final density of 0.2 g/cc. The chart is a plot of void volume generation as a function of saturation. This chart illustrates that the sample begins with a void volume capability of 6 cc/g and doubles that capability to nearly 12 cc/g as the sample reaches 7 g/g saturation.

Example 2

Figure 2 shows comparable data for a layered composite that utilizes a slow Dow AFA-130-53C superabsorbent material in place of the Stockhausen superabsorbent FAV880 from Figure 1 and the material was produced at a density of 0.055 g/cc. All other materials and pressing conditions are identical to those in Example 1. As can be seen in Figure 2, this composition generates very similar void volume capability as a function of increased saturation.

Example 3

Another embodiment is a composite with the same composition and pressing conditions as in Example 2, but the composite had an as produced density of 0.11 g/cc. With this starting density, the void volume generation is only 10 cc/g at 7 g/g

of saturation, but still falls within the scope of this invention. The void volume generation properties of this composite are illustrated in Figure 3.

Example 4

5 This embodiment is a composite with the same materials and pressing conditions as Example 1 except that 55 weight percent pulp and 40 weight percent superabsorbent were used. This composite had an as produced density of 0.05 g/cc and a final density of 0.2 g/cc. There is no Figure representing the void volume generation of this Example.

10

Example 5

 This embodiment is a composite with the same materials and pressing conditions as Example 2 except that 55 weight percent pulp and 40 weight percent superabsorbent were used. This composite had an as produced density of 0.053 g/cc and a final density of 0.2 g/cc. There is no Figure representing the void volume generation of this Example.

15

 The Tables 1 and 2 show Gravimetric Absorbancy Test (GAT) data for the five Examples as well as for commercially available Huggies® diaper material. The Huggies® diaper material was taken from the retention material below the surge material in the frontal target zone of Huggies® for Him Ultratrim® diapers, size 3. These diapers are commercially available from the Kimberly-Clark Corporation, Dallas, TX. These samples had a density of about 0.02 g/cc and contained either Stockhausen FAV880 or Dow 2035 commercially available superabsorbents in an amount of about 38 weight percent and Coosa 1654 pulp, available from Coosa Mills, Coosa, Alabama, in an amount of about 62 weight percent.

20

25

Table 1 shows the Huggies® diaper results for two sets of samples as well as results for each of the Examples with two layers, as indicated. The Huggies® diaper samples had a basis weight of about 800 gsm and the Examples with two layers had a basis weight of about 400 gsm. The results in Table 2 show the Examples at a basis weight of about 800 gsm.

As can be seen from Table 1, all of the Example materials absorbed the 10 ml insults much more quickly than the Huggies® diaper on the first and second insults even though only half the mass of material was tested. The Example materials had about half the bulk (caliper) of the Huggies® diaper material. The Example materials used the available void volume more efficiently than the commercially available material as can be seen from the void volume data on the right hand side of Table 1. The Examples maintained high intake rates even after reaching 70 percent saturation.

Table 2 shows similar data as Table 1 but uses a basis weight and starting bulk for the Example materials which is about the same as the Huggies® diaper material. As can be seen from the Table 2 data, the Example material all absorbed all three 10 ml insults far faster than the Huggies® diaper material. Void volume utilization was also far improved.

The material of the Examples, while superficially somewhat similar to the Huggies® diaper material in composition, provided strikingly better performance. This is believed to be due to the generation of void volume which occurs as the Example materials expand upon liquid contact. This expansion is driven not only by

normal superabsorbent expansion, as occurs in a Huggies® diaper, for example, but also by the expansion of the fibrous matrix in which the superabsorbent is contained. Superabsorbent gel blocking and subsequent liquid intake restriction is, therefore, eliminated or reduced in the composites of this invention. The

5 composites of this invention, therefore, may be used as surge materials, as retention materials with a surge above as in conventional Huggies® diapers, or may be used as retention materials without surge due to their superior absorbent properties.

10 Although only a few exemplary embodiments of this invention have been described in detail above, those skilled in the art will readily appreciate that many modifications are possible in the exemplary embodiments without materially departing from the novel teachings and advantages of this invention. Accordingly, all such modifications are intended to be included within the scope of this invention

15 as defined in the following claims. In the claims, means plus function claims are intended to cover the structures described herein as performing the recited function and not only structural equivalents but also equivalent structures. Thus although a nail and a screw may not be structural equivalents in that a nail employs a cylindrical surface to secure wooden parts together, whereas a screw employs a

20 helical surface, in the environment of fastening wooden parts, a nail and a screw may be equivalent structures.

TABLE 1

GRAVIMETRIC ABSORBENCY TEST - POINT SOURCE AT 4 CM POSITIVE HEAD

CODE	BULK INCHES	MASS GRAMS	DENSITY G/CC	1st inst sec SECONDS	2nd inst sec SECONDS	3rd inst sec SECONDS	FINAL MASS GRAMS	FINAL BULK INCHES	VOLUME CC'S	VOID VOL CC'S	FILLED W/ CC'S	UNFILLED W/ CC'S
HUGGIES												
1	0.141	2.59	0.20	28.82	42.41		22.75	0.425	39.53	37.61	20.16	17.45
2	0.140	2.84	0.22	48.03	54.17		22.80	0.457	42.51	40.40	19.96	20.44
3	0.142	2.53	0.19	70.45	78.2		22.35	0.445	41.39	39.51	19.82	19.69
AVG	0.141	2.65	0.20	48.43	58.26		22.63	0.442	41.14	39.17	19.88	19.19
HUGGIES												
1	0.142	2.96	0.22	48.70	54.9	41.6	32.80	0.521	48.46	46.26	29.84	16.42
2	0.150	2.41	0.17	29.80	40.8	43.4	32.40	0.445	41.39	39.60	28.99	9.61
3	0.148	2.80	0.20	33.20	26.7	24.8	33.00	0.480	44.84	42.56	30.20	12.36
AVG	0.147	2.72	0.20	37.23	41.8	36.6	32.73	0.482	44.83	42.81	30.01	12.00
Ex. 1, two layers												
1	0.075	1.55	0.222	11.20	10.91		21.70	0.301	28.00	26.90	20.15	6.75
2	0.076	1.48	0.212	17.33	11.70		21.90	0.280	28.97	25.92	20.42	5.50
3	0.073	1.54	0.227	19.33	12.03		22.00	0.270	25.11	24.02	20.46	3.56
AVG	0.074	1.52	0.220	15.95	11.55		21.87	0.287	26.69	25.61	20.34	5.27
Ex. 2, two layers												
1	0.083	1.39	0.18	20.39	23.5		20.97	0.248	22.88	21.90	19.58	2.32
2	0.080	1.28	0.17	18.9	14		20.41	0.24	22.32	21.42	19.13	2.29
3	0.080	1.39	0.19	19.6	11.7		21.42	0.265	24.85	23.66	20.03	3.63
AVG	0.081	1.35	0.18	19.63	16.4		20.93	0.250	23.78	22.32	19.58	2.74
Ex. 3, two layers												
1	0.078	1.46	0.20	24.3	15.1		21.88	0.249	23.16	22.13	20.22	1.91
2	0.078	1.39	0.19	22.1	14		21.02	0.239	22.23	21.24	19.63	1.61
3	0.078	1.39	0.19	25	12.9		20.95	0.248	23.07	22.08	19.46	2.62
AVG	0.078	1.41	0.19	23.80	14		21.18	0.245	22.82	21.82	19.77	2.05
Ex. 4, two layers												
1	0.076	1.52	0.21	20.59	11.66		31.40	0.352	32.74	31.59	29.88	1.71
2	0.078	1.41	0.19	23.67	9.75		32.80	0.356	33.11	32.04	31.39	0.65
3	0.083	1.47	0.19	16.10	7.97		31.00	0.352	32.74	31.62	30.43	1.19
AVG	0.080	1.47	0.20	20.12	9.79		32.03	0.353	32.66	31.75	30.57	1.19
Ex. 4, two layers												
1	0.074	1.54	0.224	19.77	8.09	14.40	32.60	0.360	35.34	34.17	31.08	3.11
2	0.076	1.29	0.178	15.41	11.77	16.12	31.10	0.360	33.48	32.50	29.81	2.69
3	0.078	1.42	0.198	18.76	9.53	18.50	30.60	0.360	33.48	32.41	29.18	3.23
AVG	0.077	1.42	0.199	17.96	9.60		31.43	0.367	34.10	33.03	30.02	3.01
Ex. 5, two layers												
1	0.075	1.41	0.202	16.74	7.69		21.08	0.295	27.44	26.37	20.55	6.82
2	0.074	1.39	0.202	21.07	6.91		21.50	0.293	27.25	26.20	20.11	6.09
3	0.078	1.24	0.169	14.74	5.84		20.80	0.240	22.32	21.36	19.68	1.82
AVG	0.076	1.35	0.181	16.18	7.48		21.42	0.276	25.07	24.65	20.07	4.50

TABLE 2

GRAVIMETRIC ABSORBENCY TEST - POINT SOURCE AT 4 CM POSITIVE HEAD

CODE	BULK INCHES	MASS GRAMS	DENSITY G/CC	1st instl sec SECONDS	2nd instl sec SECONDS	3rd instl sec SECONDS	FINAL MASS GRAMS	FINAL BULK INCHES	VOLUME CC'S	VOID VOL CC'S	FILLED VOL CC'S	UNFILLED VOL CC'S
Ex. 1, four layers												
1	0.152	2.73	0.193	13.10	7.55	8.39	33.00	0.465	43.25	41.18	30.27	10.91
2	0.143	2.97	0.223	16.15	9.16	9.62	32.30	0.517	48.09	45.84	29.33	16.51
3	0.141	3.01	0.230	13.10	7.60	10.13	33.50	0.510	47.43	45.15	30.49	14.86
AVG	0.145	2.90	0.215	14.12	8.10		32.93	0.497	46.26	44.08	30.03	14.03
Ex. 2, four layers												
1	0.153	3.02	0.21	17.30	12.3	13.50	33.30	0.452	42.04	39.90	30.28	9.62
2	0.150	2.95	0.21	15.90	8.7	11.10	32.80	0.467	43.44	41.35	29.85	11.50
3	0.140	2.77	0.21	18.00	9.9	10.70	32.80	0.431	40.09	38.13	30.03	8.10
AVG	0.148	2.91	0.21	17.07	10.3	11.77	32.97	0.450	41.85	39.79	30.05	9.74
Ex. 3, four layers												
1	0.139	2.97	0.23	19.90	8.6	12.00	33.30	0.450	41.85	39.75	30.33	9.42
2	0.135	2.97	0.24	19.50	9.8	12.20	33.40	0.452	42.04	39.94	30.43	9.51
3	0.153	2.98	0.21	19.60	8.8	12.50	33.50	0.450	41.85	39.74	30.52	9.22
AVG	0.142	2.97	0.22	19.67	8.4	12.23	33.40	0.451	41.92	39.81	30.43	9.38
Ex. 4, four layers												
1	0.142	3.05	0.23	16.20	12.1	12.00	33.10	0.637	59.25	56.93	30.05	26.88
2	0.155	2.75	0.19	16.70	12.2	10.40	32.80	0.572	53.20	51.12	30.05	21.07
3	0.153	2.66	0.20	15.90	18.3	28.60	32.40	0.593	55.15	52.99	29.54	23.45
AVG	0.150	2.89	0.21	16.27	14.2	17.00	32.77	0.601	55.87	53.68	29.88	23.80
Ex. 5, four layers												
1	0.152	2.65	0.187	16.42	8.49	9.63	32.60	0.560	52.09	50.08	29.85	20.13
2	0.154	2.77	0.193	16.05	10.43	10.42	32.00	0.520	48.37	46.28	29.23	17.03
3	0.158	2.77	0.189	14.27	13.08	17.40	32.50	0.515	47.80	45.80	28.73	16.07
AVG	0.155	2.73	0.190	15.58	10.66		32.37	0.532	49.45	47.38	29.64	17.74

What is claimed is:

1. An expandable absorbent composite comprising pulp in an amount from about 30 to about 90 weight percent, superabsorbent material in an amount from about 10 to about 70 weight percent and a binder in an effective amount,
5 wherein said composite has been compressed to a density of from about 0.1 g/cc to about 0.3 g/cc.
2. The composite claim 1 having pulp in an amount from about 40 to about 80 weight percent, superabsorbent in an amount from about 10 to about 50 weight percent and binder fiber in an amount from about 2 to about 10 weight percent.
- 10 3. The composite claim 1 having pulp in an amount from about 55 to about 75 weight percent, superabsorbent in an amount from about 20 to about 40 weight percent and binder fiber in an amount from about 3 to about 8 weight percent.
4. An expandable absorbent composite which has been compressed and which expands rapidly when wetted to greater than 80% of its uncompressed thickness
15 and to greater than 90% of its uncompressed thickness when saturated.
5. A personal care product selected from the group consisting of diapers, training pants, feminine hygiene products, absorbent underpants and adult incontinence products comprising the material of claim 1.
6. The product of claim 5 wherein said personal care product is an adult
20 incontinence product.
7. The product of claim 5 wherein said personal care product is a feminine hygiene product.
8. The product of claim 5 wherein said personal care product is a diaper.
9. A process of producing an expandable absorbent composite comprising the
25 steps of:

blending together pulp in an amount from about 28 to about 90 weight percent, superabsorbent material in an amount from about 8 to about 70 weight percent and a binder fiber in an amount from about 2 to about 20 weight percent to form a web;

- 5 compressing said web at a temperature and time sufficient to produce a stabilized composite having a density between about 0.1 g/cc and about 0.3 g/cc.

10. The process of claim 9 wherein said web is heated at a temperature between about 80 and 100 °C.

1/ 3

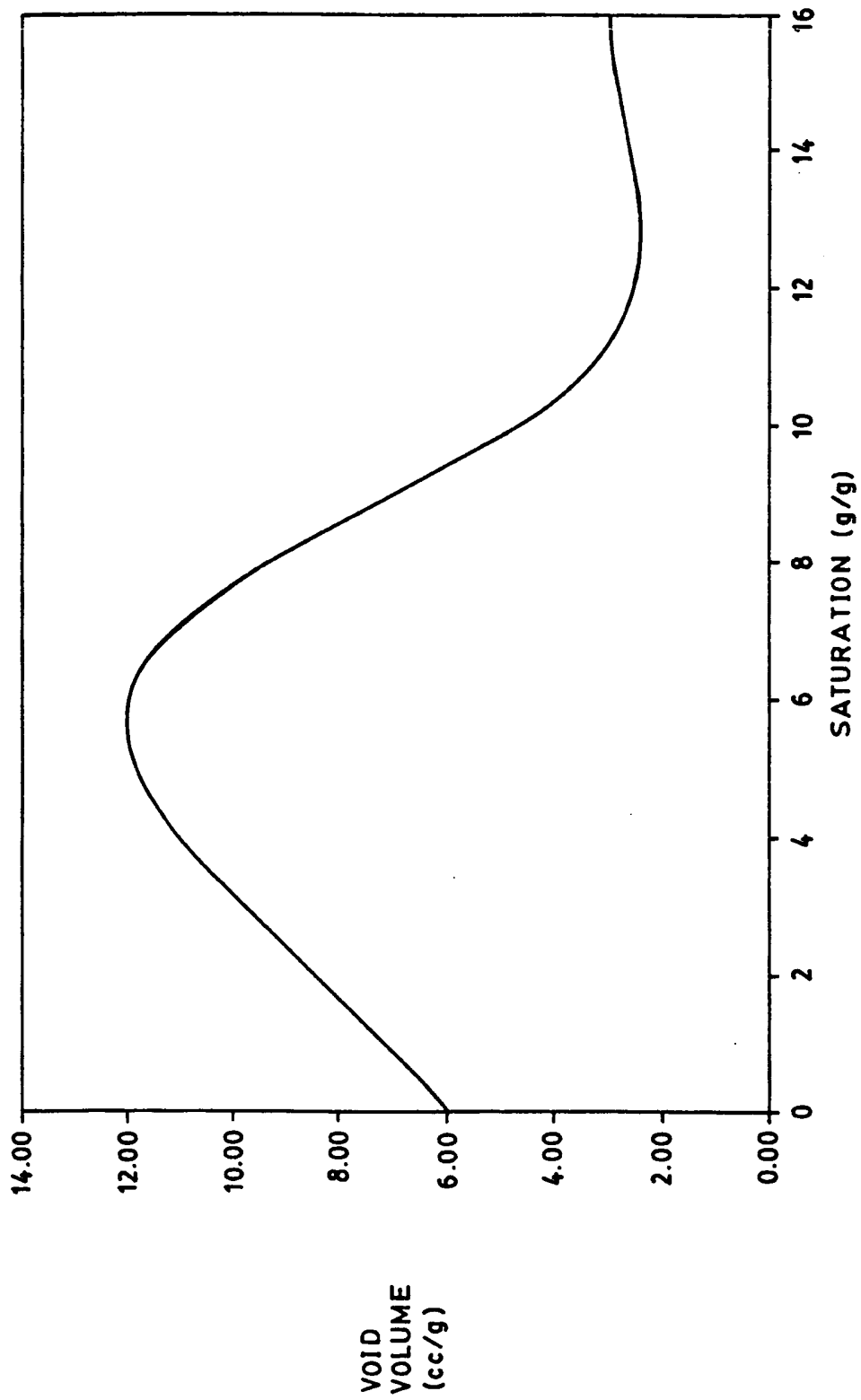
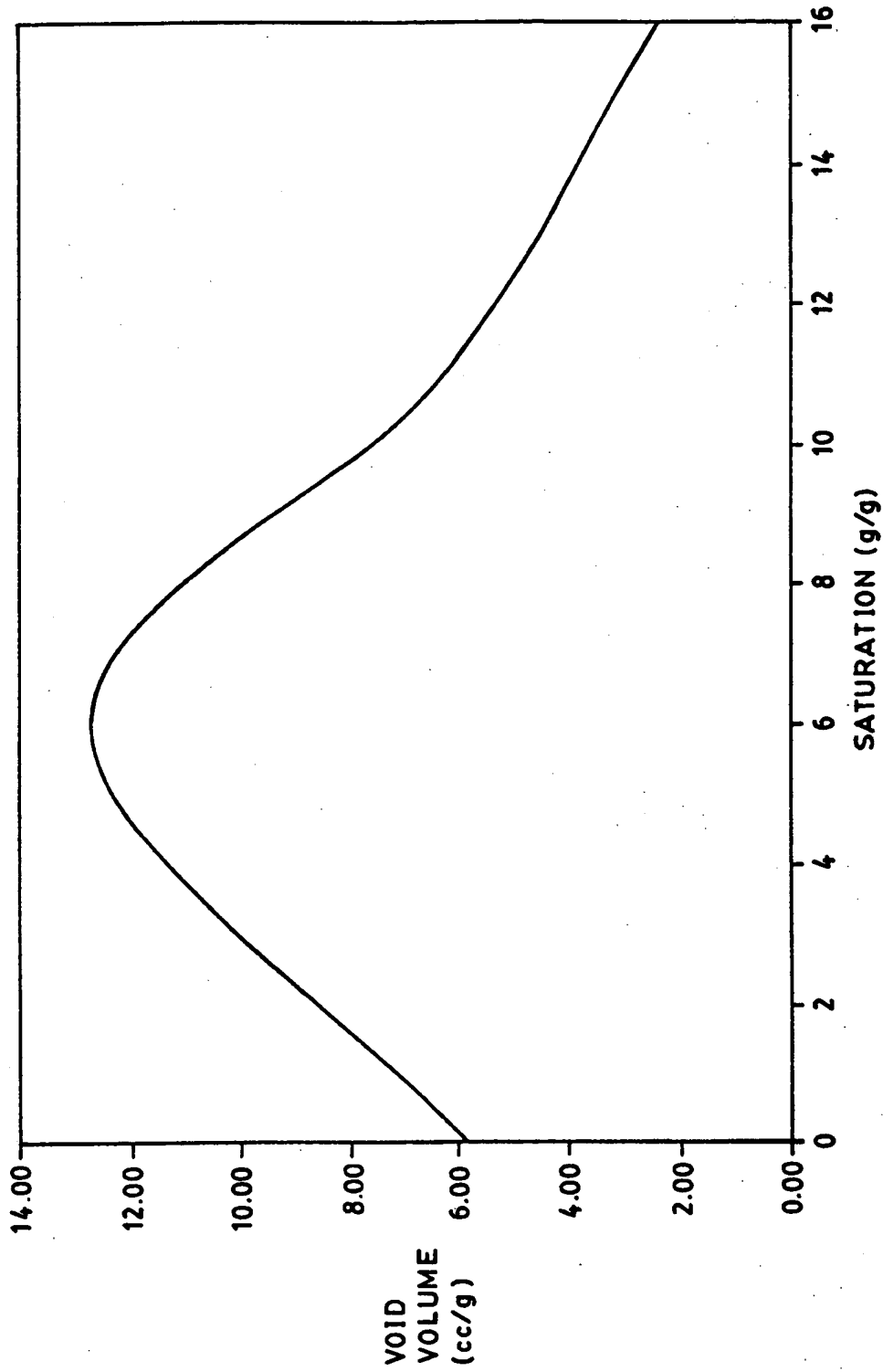


FIG. 1

2 / 3

**FIG. 2**

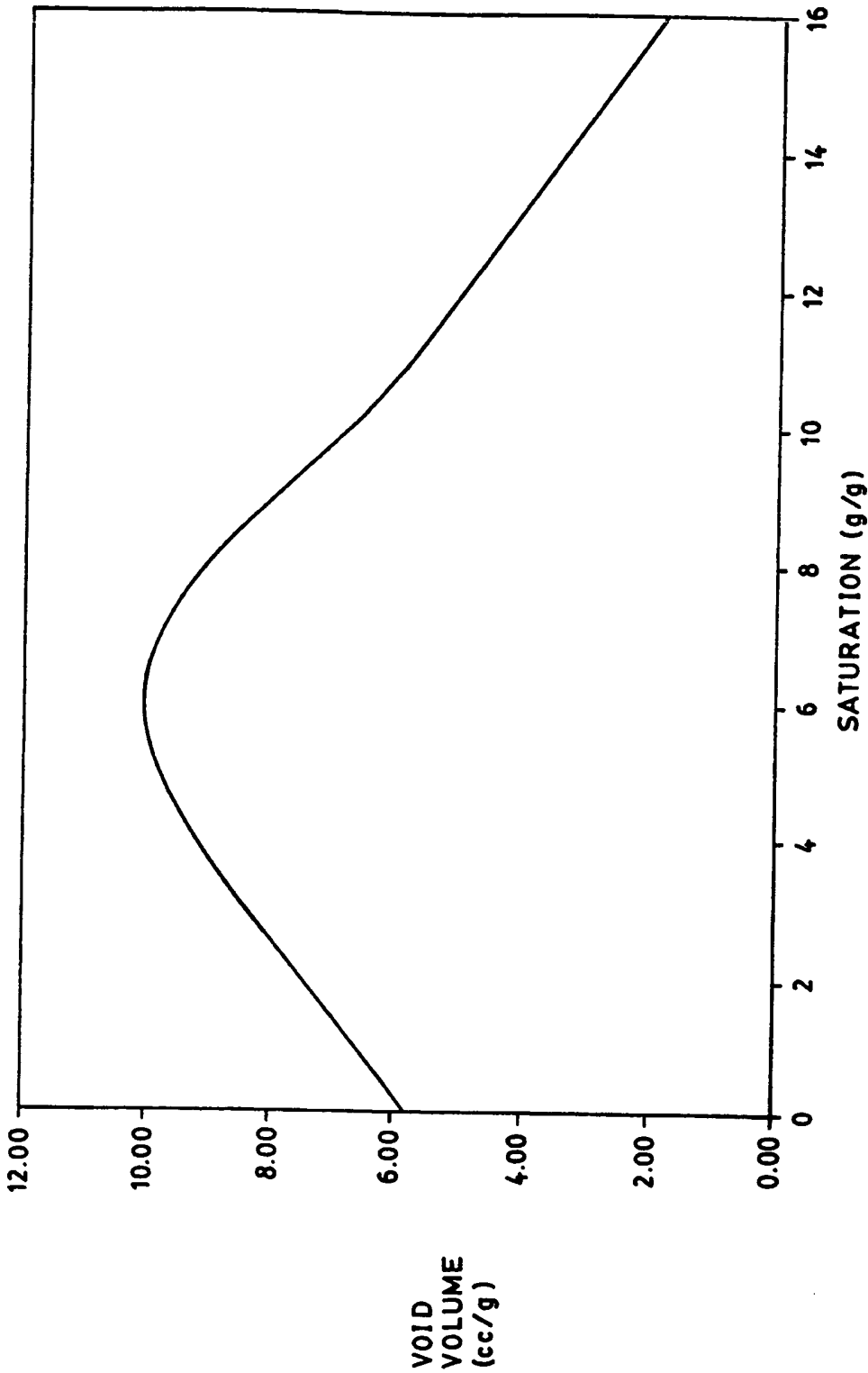


FIG. 3

INTERNATIONAL SEARCH REPORT

Int. Application No

PCT/US 98/26027

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 A61L15/60 A61F13/15

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A61L A61F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>WO 96 31644 A (WEYERHAEUSER CO) 10 October 1996 see page 9, line 8 - line 15 see page 14, line 24 - page 15, line 3 see page 15, line 18 - page 17, last line see page 19, line 11 - line 14 see page 20, line 24 - page 22, line 22 see page 24, line 28 - page 25, line 2 see page 27, line 29 - page 28, line 23 see page 33; table 3 see claims</p> <p style="text-align: center;">--- -/--</p>	1-9

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

1 April 1999

Date of mailing of the international search report

12/04/1999

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Thornton, S

INTERNATIONAL SEARCH REPORT

Int'l Application No
PCT/US 98/26027

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>WO 96 27703 A (WEYERHAEUSER CO ;HALABISKY DONALD D (US); WEST HUGH (US); HAJNAL A) 12 September 1996 see page 7, line 3 - page 9, line 23 see page 10, line 24 - page 11, line 5 see page 12, line 30 - page 14, line 28 see page 19, table see page 26, line 25 - line 27 see page 27, line 19 - page 28, line 2 see claims</p> <p style="text-align: center;">---</p>	1-3,5-10
A	<p>EP 0 712 659 A (NIPPON CATALYTIC CHEM IND) 22 May 1996 see page 2, line 47 - page 3, line 18 see page 4, line 44 - page 5, line 3 see claims</p> <p style="text-align: center;">---</p>	1-3,5-9
A	<p>EP 0 615 736 A (KIMBERLY CLARK CO) 21 September 1994 see page 5, line 44 - line 50 see page 6, line 2 - line 6 see page 14, line 41 - line 47 see figures 1-4 see claims</p> <p style="text-align: center;">---</p>	1-3,5-8
A	<p>WO 96 21409 A (PROCTER & GAMBLE) 18 July 1996 see page 8, line 18 - line 34 see page 16, line 13 - line 26 see page 17, line 23 - line 34 see page 18, line 13 - line 17 see page 21, line 6 - line 33 see claims</p> <p style="text-align: center;">---</p>	1,4-8
A	<p>WO 97 36955 A (SCHMIDT MATTIAS ;PALUMBO GIANFRANCO (DE); PROCTER & GAMBLE (US)) 9 October 1997 see page 6, line 16 - line 27 see page 11, line 17 - page 12, line 21 see page 20, line 20 - line 34 see page 22, line 21 - page 23, line 2 see page 24, line 6 - line 17 see figure 1 see claims 1,5,8,11,13,15-17,19,20</p> <p style="text-align: center;">---</p>	1,4-8
A	<p>US 5 372 877 A (KANNANKERIL CHARLES P) 13 December 1994 see column 1, line 36 - line 45 see column 2, line 5 - line 39 see column 2, last line - column 3, line 2 see column 4, line 66 - column 5, line 9 see examples see claims 1-11</p> <p style="text-align: center;">-----</p>	1-10

INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/US 98/26027

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9631644 A	10-10-1996	US 5807364 A CA 2191429 A EP 0769082 A JP 10501593 T	15-09-1998 10-10-1996 23-04-1997 10-02-1998
WO 9627703 A	12-09-1996	AU 5183596 A CA 2213449 A EP 0813626 A US 5837627 A	23-09-1996 12-09-1996 29-12-1997 17-11-1998
EP 0712659 A	22-05-1996	US 5760080 A WO 9534377 A JP 8057311 A JP 8057310 A	02-06-1998 21-12-1995 05-03-1996 05-03-1996
EP 0615736 A	21-09-1994	AU 4691097 A AU 5632794 A BR 9400651 A CA 2114815 A CN 1095622 A JP 6254118 A MX 9301105 A PL 302369 A US 5601542 A ZA 9400576 A	12-02-1998 01-09-1994 27-09-1994 25-08-1994 30-11-1994 13-09-1994 31-08-1994 05-09-1994 11-02-1997 13-09-1994
WO 9621409 A	18-07-1996	AU 4897296 A BR 9606738 A CA 2209133 A CN 1176592 A CZ 9702178 A EP 0802775 A FI 972916 A HU 9800806 A JP 10512167 T NO 973188 A US 5817081 A	31-07-1996 30-12-1997 18-07-1996 18-03-1998 12-11-1997 29-10-1997 09-09-1997 28-08-1998 24-11-1998 10-09-1997 06-10-1998
WO 9736955 A	09-10-1997	EP 0797966 A AU 2536397 A CA 2250138 A	01-10-1997 22-10-1997 09-10-1997
US 5372877 A	13-12-1994	NONE	

THIS PAGE BLANK (USPTO)

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☒ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☒ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.

THIS PAGE BLANK (USPTO)